



4-30-1965

The Synthesis and Reactions of 8-Carboxy Isatoic Anhydride

James E. Scheirer
Ursinus College

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The Synthesis and Reactions
of 8 - Carboxy Isatoic Anhydride

James E. Scheirer
Department of Chemistry
Ursinus College
30 April 1965

This paper is submitted to the faculty of Ursinus
College in partial fulfillment of the requirements
for departmental honors in chemistry.

Submitted by:

James E. Scheirer

Approved by:

Noga P. Staiger

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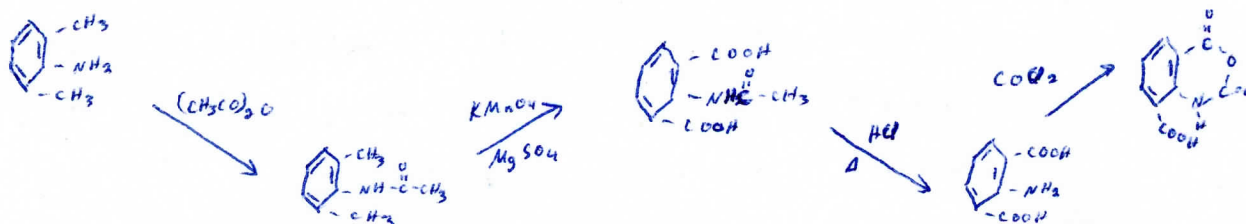
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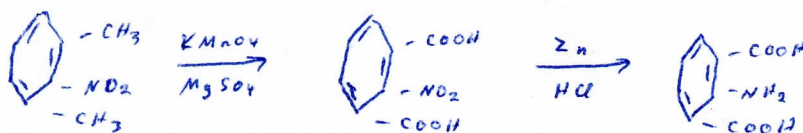
Introduction

The object of this research project was the preparation and characterization of 8 - carboxy isatoic anhydride and the study of its reactions, leading to the synthesis of compounds of new composition.

The proposed path of the synthesis was the acetylation of 2,6 dimethyl aniline, oxidation to 2-acetamino isophthalic acid, and ring closure with phosgene to form the desired 8 - carboxy isatoic anhydride. The route is summarized as follows:



The preparation of 2-amino isophthalic acid has been reported in the literature, but it is not commercially available. It can be prepared from 2,6 dimethyl aniline¹ as shown above or from 2,6 dimethyl nitrobenzene² by oxidation to 2-nitro isophthalic acid followed by reduction to 2-amino isophthalic acid as illustrated below:



Since both of these are commercially available and relatively inexpensive, either one could have been chosen as the starting material. At first 2,6 dimethyl nitrobenzene was used, but several attempts at oxidation led only to the recovery of inorganic salts. For this reason the route using 2,6 dimethyl aniline was tried, and, when this synthesis was successful, it was used exclusively.

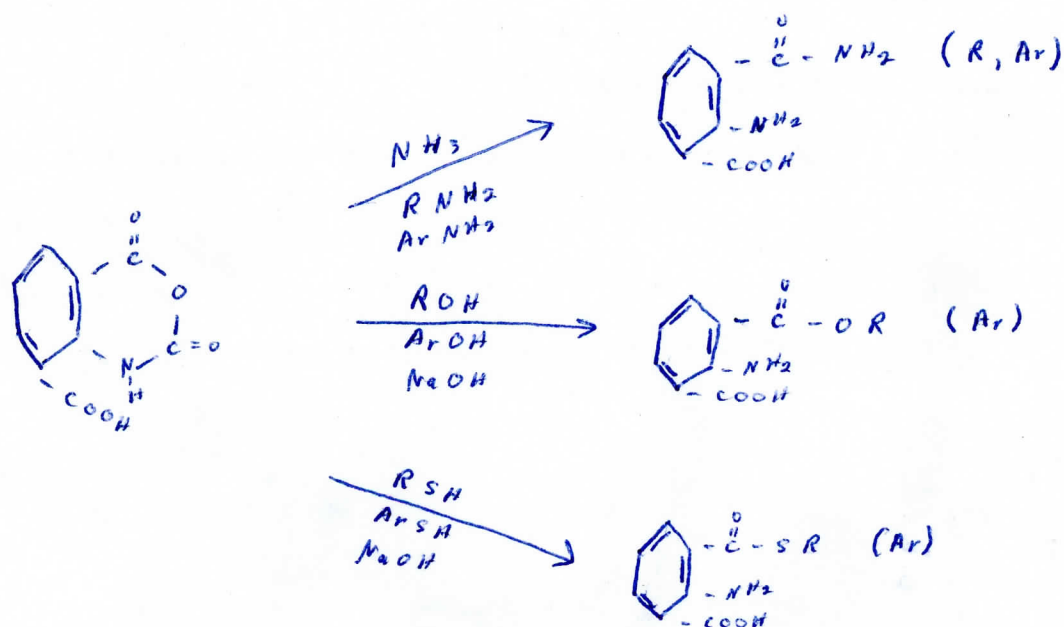
It was further proposed that 8 - carboxy isatoic anhydride might undergo a further reaction with phosgene. The hydrogen atom attached to the nitrogen atom in isatoic anhydride is labile. Thus if it should react with phosgene a second ring closure would be obtained as shown in the following scheme:



However this secondary reaction was not detected at any time, and the product formed would probably be unstable.

After the synthesis of 8 - carboxy isatoic anhydride had been confirmed, its reactions were studied. When attacked by nucleophilic reagents, 8 - carboxy isatoic anhydride undergoes ring cleavage in a manner similar to that in unsubstituted isatoic anhydride. 8 - carboxy isatoic anhydride reacts with

ammonia and aliphatic and aromatic amines to form 2-amino-3-carboxy benzamides; with alcohols and phenols to form 2-amino-3-carboxy benzoates; and with mercaptans and thiophenols to form 2-amino-3-carboxy thio benzoates. These reactions are outlined in the following scheme:



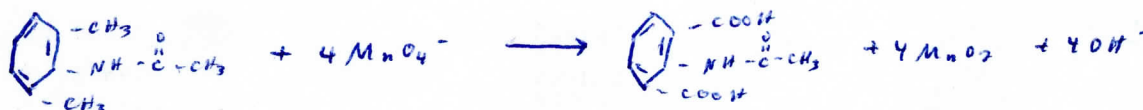
These products are all compounds of new composition and are characterized for physical properties and confirmed by analysis. Infrared spectra are available for each compound.

Synthesis of 8 - Carboxy Isatoic Anhydrides

Preparation of 2-acetamino isophthalic acid

2-acetamino isophthalic acid was prepared by the oxidation of 2,6 dimethyl aniline using potassium permanganate in a magnesium sulfate heptahydrate buffer solution. This is the method used by Bogert and Kropff³ with slight modifications. The magnesium sulfate buffer keeps the solution slightly alkaline. This method is described for small samples (5 grams of acetylated amine in 100 ml of water), but it has been found to work better when larger samples of acetylated amine are used while the volume of water is kept constant.⁴ For this reason 25 gram samples were used.

Equation:



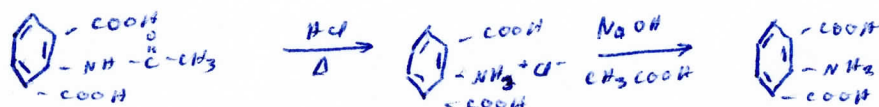
Experimental: 25 grams of 2,6 dimethyl aniline is acetylated with excess acetic anhydride to form 2-acetamino m-xylene. Nearly 100% yield is obtained. The 2-acetamino m-xylene, 50 grams of potassium permanganate, and 100 grams of magnesium sulfate heptahydrate are suspended in about 250 ml of water in a 2 liter beaker. This reaction mix is heated on a hotplate - stirrer set on low heat until the reaction begins (about 80°C).

The reaction immediately becomes vigorous and the beaker must be removed from the hotplate. After the reaction has subsided, a second 50 grams of potassium permanganate is added and the mix is again heated to 80°C and removed from the hotplate. When the reaction has stopped, the sides of the beaker are washed down with water and the mixture is heated with stirring until no pink color remains. The mix is cooled in an ice bath and made strongly basic with 20% sodium hydroxide. The sludge is stirred well and suction filtered. It is washed several times with 20% sodium hydroxide and the filtrates are saved. Finally the manganese dioxide sludge is removed from the filter, stirred vigorously with 20% sodium hydroxide, and filtered. The filtrate contains colloidal manganese hydroxide which must be removed by gravity filtration. The filtrate is now acidified with acetic acid and since the precipitation is slow, it is allowed to stand overnight. A fine white powder is obtained when the mix is suction filtered and dried. After recrystallization from methanol, the yield is about 7 grams, which is about 19% of the theoretical. The melting point was observed to be 206-208°C. The literature value is 205-206°C.⁵ Infrared spectrum of 2-acetamino isophthalic acid: Experiment #556, III, 2/9/65

Preparation of 2-amino isophthalic acid

The hydrolysis of 2-acetamino isophthalic acid can be effected in either acidic or basic medium. Both methods were tried on small samples and it was found that acidic hydrolysis was more effective.

Equation:

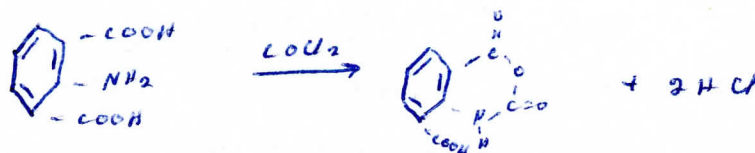


Experimental: 17.6 grams of 2-acetamino isophthalic acid is placed in a beaker containing 200 ml. of concentrated hydrochloric acid. The mix is boiled for one-half hour on a hot-plate - stirrer and then cooled in an ice bath. The amine hydrochloride is filtered off, dissolved in 200 ml of water, and made basic with 10% sodium hydroxide. The solution is acidified with acetic acid and allowed to stand overnight. Nearly a 100% yield is obtained of the white powder, which decomposes at 270°C . The literature predicts decomposition above 260°C .⁶ Infrared spectrum of 2-amino isophthalic acid: Experiment #556, IV, 2/23/65

Synthesis of 8 - Carboxy Isatoic Anhydride

Ring closure of 2-amino isophthalic acid to produce 8 - carboxy isatoic anhydride was effected by phosgene, using dioxane as a solvent.⁷

Equation:

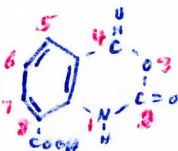


Experimental: One gram of 2-amino isophthalic acid is made into a slurry with 10 ml of dioxane in a 50 ml round-bottom flask. Under a closed, well-ventilated hood the solution is saturated with phosgene gas, and then refluxed for one-half hour. The solution is then poured into an ice-water mixture and the precipitate is filtered off. The fluffy white powder is recrystallized from dioxane. Upon drying and exposure to air the 8 - carboxy isatoic anhydride turns yellow, with a slight green tinge in sunlight - and displays blue fluorescence in solution. The yield is about 40-50%, and the compound decomposes slowly at 255-257 , turning dark red with evolution of a gas. The analysis was as follows: theoretical carbon 52.18%, hydrogen 2.43%, nitrogen 6.76%; found carbon 51.96%, hydrogen 2.58%, nitrogen 6.54%. Infrared spectrum of 8 - carboxy isatoic anhydride: Experiment #556, V, 3/16/65

Reactions of 8 - Carboxy Isatoic Anhydride

In 8 - carboxy isatoic anhydride both the number two and the number four carbon atoms are susceptible to nucleophilic attack, due to the electron withdrawal of the carbonyl oxygen atoms.

However the number two



carbon atom is not as susceptible to attack because the high electron density of the adjacent nitrogen atom reduces the positive character of the number two carbon atom. Thus the predominant reaction involves the attachment of a nucleophile to the number four carbon atom and the evolution of carbon dioxide.

8 - carboxy isatoic anhydride undergoes the same reactions as unsubstituted isatoic anhydride, but the reactions are slower and require more vigorous conditions. It reacts readily with ammonia and amines to form amides, although the reaction is not as fast as with unsubstituted isatoic anhydride. Formation of esters and thio esters was much slower than in unsubstituted isatoic anhydride.

Synthesis of 2-amino-3-carboxy Benzanilide

Experimental: 0.5 grams of 8 - carboxy isatoic anhydride is mixed

with 5 ml of dioxane to form a slurry and 0.5 ml of aniline is added. The reaction mixture is refluxed for 3/4 hour. The solution is cooled and, when no precipitate is obtained, some of the solvent is boiled off under reduced pressure until a pale yellow precipitate is obtained. This product is recrystallized from methanol.

2-amino-5-carboxy benzanilide is a pale yellow powder melting at 247-248°C with decomposition. The analysis is as follows: theoretical carbon 65.63%, hydrogen 4.72%; found carbon 65.43%, hydrogen 4.80%. Infrared spectrum: Experiment #556, VI, 3/23/65.

Synthesis of 2-amino-3-carboxy Methyl Benzoate

Experimental: One gram of 3 - carboxy isatoic anhydride is suspended in 10 ml of methanol and a tiny chip of sodium hydroxide is added as a catalyst. The mix is refluxed for five hours, cooled in an ice bath, and the white crystalline solid is filtered off. The product is recrystallized from methanol.

2-amino-3-carboxy methyl benzoate is a white solid which melts at 174-175°C. The analysis is as follows: theoretical carbon 55.38%, hydrogen 4.65%; found carbon 55.42 %, hydrogen 4.73 %. Infrared spectrum: Experiment #556, VII, 4/22/65.

Synthesis of 2-amino-3-carboxy N-hexyl Benzamide

Experimental: One gram of 8 - carboxy isatoic anhydride is placed in a 50 ml beaker and n-hexylamine is added dropwise until no further evolution of carbon dioxide is noted. The solution is acidified with acetic acid and the white precipitate is filtered off. The product is recrystallized from hot water.

2-amino-3-carboxy N-hexyl benzamide is a white powder which melts at 181-182°C. The analysis is as follows: theoretical carbon 63.64%, hydrogen 7.58%; found carbon 63.59%, hydrogen 7.62 %. Infrared spectrum: Experiment #556, VIII, 4/23/65.

Synthesis of 2-amino-3-carboxy Benzamide

Experimental: 0.5 grams of 8 - carboxy isatoic anhydride is placed in a 50 ml beaker and concentrated ammonia is added dropwise until no more carbon dioxide is evolved. The solution is acidified with acetic acid and the white precipitate is filtered off. When recrystallized from hot water the product becomes yellow, but on drying under a heat lamp for one-half hour it becomes white again.

2-amino-3-carboxy benzamide is a white powder which melts at 246-247°C. Three sets of analyses were obtained for this compound. They are as follows:

	Theoretical	Found		
C	53.33	52.09	50.16	53.54
H	4.47	4.65	4.55	4.49
N	15.55	15.05		

Infrared spectrum: Experiment #556, IX A, 4/13/65 and IX B, 4/28/65

Synthesis of 2-amino-3-carboxy Thio Phenyl Benzoate

Experimental: One gram of 8-carboxy isatoic anhydride is mixed with 10 ml of dioxane to form a slurry and 5 ml of thiophenol are added. A tiny chip of sodium hydroxide is used as a catalyst and the reaction mix is refluxed for one-half hour, a reaction time found suitable for 6-⁸ and 7-⁹ carboxy isatoic anhydride. The solution is cooled in an ice bath and the yellow precipitate is recrystallized from methanol. The results of analysis are as follows:

	Theoretical thio ester	Theoretical 8 carboxy IA	Found
C	61.54	52.18	57.83
H	4.03	2.43	2.47

The precipitate is believed to consist mainly of recovered 8 - carboxy isatoic anhydride. Infrared spectrum: Experiment #556, X A, 4/14/65

A second one gram sample of 8 - carboxy isatoic anhydride was treated in the same manner, except that it was refluxed for 6 hours. After cooling in an ice bath, a yellow precipitate of unreacted isatoic anhydride is filtered off. Upon addition of a small amount of water, a yellow oil is obtained which becomes crystalline when the dioxane is boiled off. The precipitate is recrystallized from methanol.

2-amino-3-carboxy thio phenyl benzoate is a yellow powder which melts at 234-235°C. The analysis is as follows: theoretical carbon, 61.54%, hydrogen 4.03%; found carbon 61.86%, hydrogen 4.15%. Infrared spectrum: Experiment #556 X B, 4/27/65

Synthesis of 2-amino-3-carboxy Phenyl Benzoate

Experimental: One gram of 8 - carboxy isatoic anhydride is mixed with 10 ml of dioxane to form a slurry and 5 grams of phenol are added. A tiny chip of sodium hydroxide is used as a catalyst and the mix is refluxed for one hour. The solution is cooled in an ice bath and the white solid is filtered off and dried. The results of analysis are as follows:

	Theoretical ester	Theoretical 8-carboxy IA	Found
C	65.37	52.18	52.08
H	4.28	2.43	2.54

This precipitate can be seen to consist of recovered 8 - carboxy isatoic anhydride. Infrared spectrum: Experiment #556 XI A, 4/14/65

A second one gram sample of 8 - carboxy isatoic anhydride was treated in the same manner, except that it was refluxed for 9 hours. After cooling in an ice bath a white precipitate is obtained, which is recrystallized from methanol.

2-amino-3-carboxy phenyl benzoate is a light yellow powder when recrystallized from methanol. It melts at 254-255°C. The analysis is as follows: theoretical carbon 65.37%, hydrogen 4.28%; found carbon 65.18 %, hydrogen 4.20 %. Infrared spectrum: Experiment #556, XI B, 4/2/65

Synthesis of 2-amino-3-carboxy Hexylthio Benzoate

Experimental: 1 gram of 8 - carboxy isatoic anhydride is dissolved in 10 ml of dioxane and 5 ml of hexanethiol is added. A tiny chip of sodium hydroxide is added as a catalyst and the mix is refluxed for one hour. Upon cooling, a yellow precipitate is obtained, which is identified from infrared spectra as recovered 8 - carboxy isatoic anhydride. For this reason no sample was sent for analysis and no further attempts were made to prepare the compound. Infrared spectrum: Experiment #556, XII, 4/23/65

SUMMARY

1. 8 - carboxy isatoic anhydride is prepared by the acetylation of 2,6 diemthyl aniline, oxidation by potassium permanganate to 2-acetamino isophthalic acid, acid hydrolysis to form 2-amino isophthlaic acid, and ring closure with phosgene in dioxane to form the desired compound.
2. 8 - carboxy isatoic anhydride is attacked by common nucleophiles as is unsubstituted isatoic anhydride, but the 8 - carboxy isatoic anhydride is less reactive than the unsubstituted compound.

Footnotes

1. Beilsteins Handbuch der Organische Chemie, XII, 1109.
2. I bid, IX, 839.
3. Bogert and Kropff, "On Some Amino and Nitro Derivatives of Benzoic, Metatoluic, and Metaphthalic Acids," Journal of the American Chemical Society, July 1909, 841
4. Unpublished works of Jacqueline I. Kroschwitz, Experiment #548
5. Heilbron, Dictionary of Organic Compounds, I, 101
6. Beilstein, XIV, 555
7. Unpublished works of Roger P. Staiger, Experiment #487
8. Kroschwitz, Experiment #548
9. Unpublished works of Elsa M. Janle, Experiment # 525

Acknowledgement

The author gratefully acknowledges the assistance of the entire Chemistry Department and especially Dr. Staiger in the preparation of this paper.

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Laboratories, Inc.

QUANTITATIVE ORGANIC MICROANALYSES

P. O. Box 4187

KNOXVILLE, TENNESSEE 37921

PHONE 546-1335
AREA CODE 615

Dr. Roger P. Staiger
Department of Chemistry
Ursinus College
Collegeville, Pennsylvania

March 25, 1965

Received: March 22nd

Dear Dr. Staiger:

Analysis of your compounds gave the following results:

Your #,	My #,	% C,	% H,	% N,
2 amino naphthalene acid monamide 557 A Ther	L-7066	52.09 53.33	4.65 4.47	15.05 15.55
carbazoyl I A 557 B Ther	L-7067	51.96 52.18	2.58 2.43	6.54 6.76

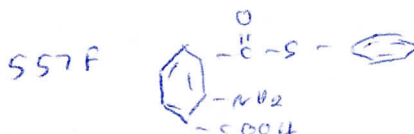
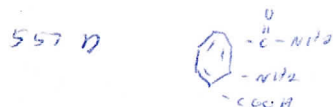
Sincerely yours,

GALBRAITH LABORATORIES, INC.

Harry W. Galbraith

Harry W. Galbraith
President

HWG:eh



GALBRAITH

Laboratories, Inc.

QUANTITATIVE ORGANIC MICROANALYSES

P. O. Box 4187

KNOXVILLE, TENNESSEE 37921

PHONE 546-1335
AREA CODE 615

Dr. R. P. Staiger
Department of Chemistry
Ursinus College
Collegeville, Pennsylvania

April 22, 1965

Received: April 19th

Dear Dr. Staiger:

Analysis of your compounds gave the following results:

Your #,	My #,	% C,	% H,	% N,
551	L-8960	46.46 <i>49.28</i>	2.56 <i>1.64</i>	10.72 <i>9.84</i>
<i>O. K.</i> 557C	L-8961	65.43 <i>65.63</i>	4.80 <i>4.79</i>	
557D	L-8962	50.16 <i>53.33</i>	4.55 <i>4.47</i>	<i>Amide</i>
557E	L-8963	52.08 <i>65.37</i>	2.54 <i>4.28</i>	<i>Phenyl Benzate</i>
557F	L-8964	57.83 <i>61.54</i>	2.47 <i>4.03</i>	<i>triphenyl benzate</i>

Sincerely yours,

GALBRAITH LABORATORIES, INC.

Harry W. Galbraith

Harry W. Galbraith
President

HWG:eh

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KNOXVILLE, TENNESSEE 37921

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AREA CODE 615

Dr. Roger P. Staiger
Department of Chemistry
Ursinus College
Collegeville, Pennsylvania

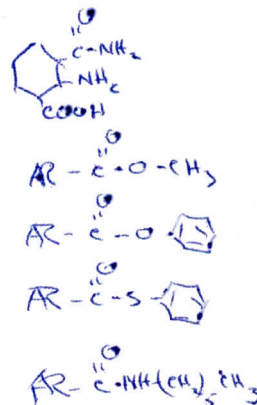
May 5, 1965

Received: May 3rd

Dear Dr. Staiger:

Analysis of your compounds gave the following results:

Your #,	My #,	% C,	% H,
557G	L-9783	53.54 53.33	4.49 4.47
557H	L-9784	55.42 55.38	4.73 4.65
557I	L-9785	65.18 65.37	4.20 4.28
557J	L-9786	61.36 61.54	4.18 4.03
557K	L-9787	63.52 63.64	7.62 7.58



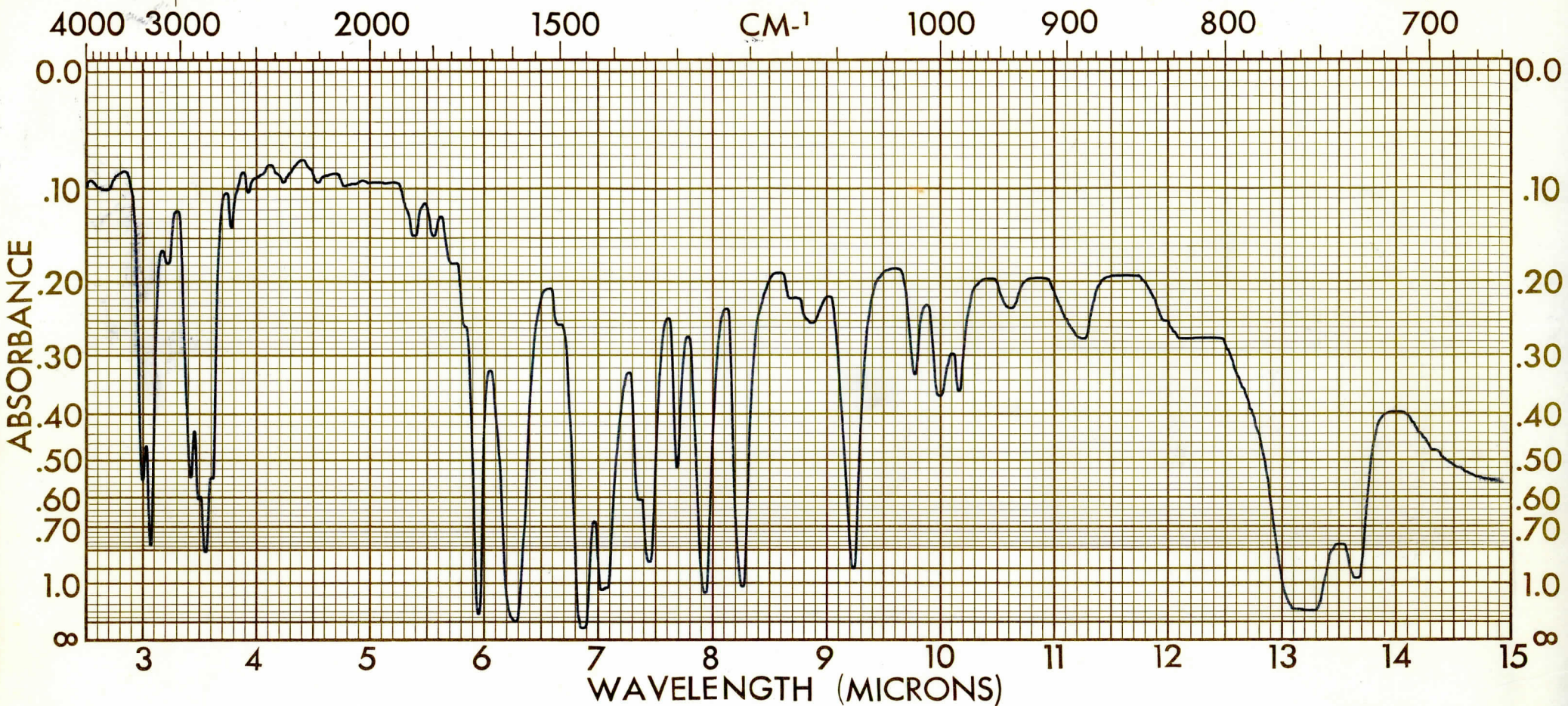
Sincerely yours,

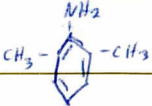
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Harry W. Galbraith

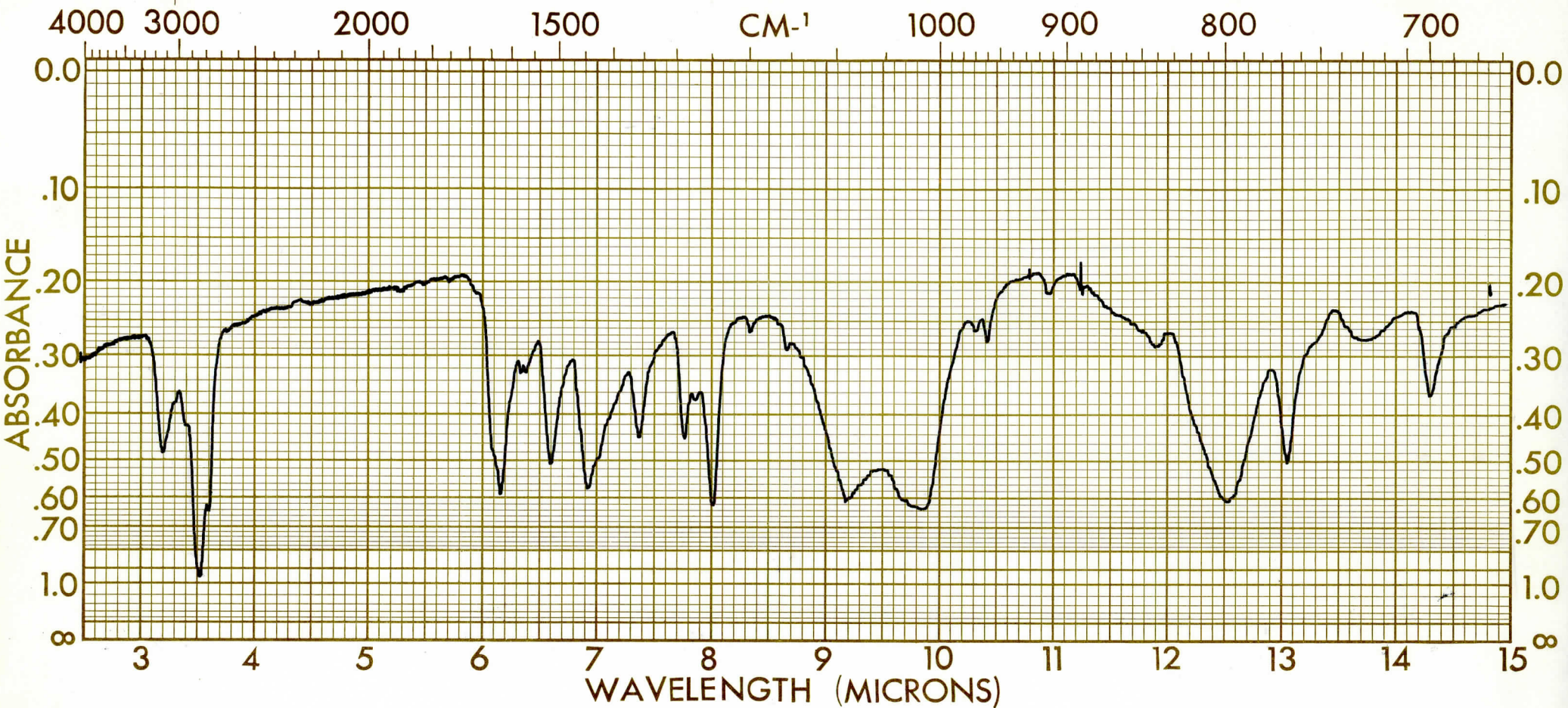
Harry W. Galbraith
President

HWG:eh



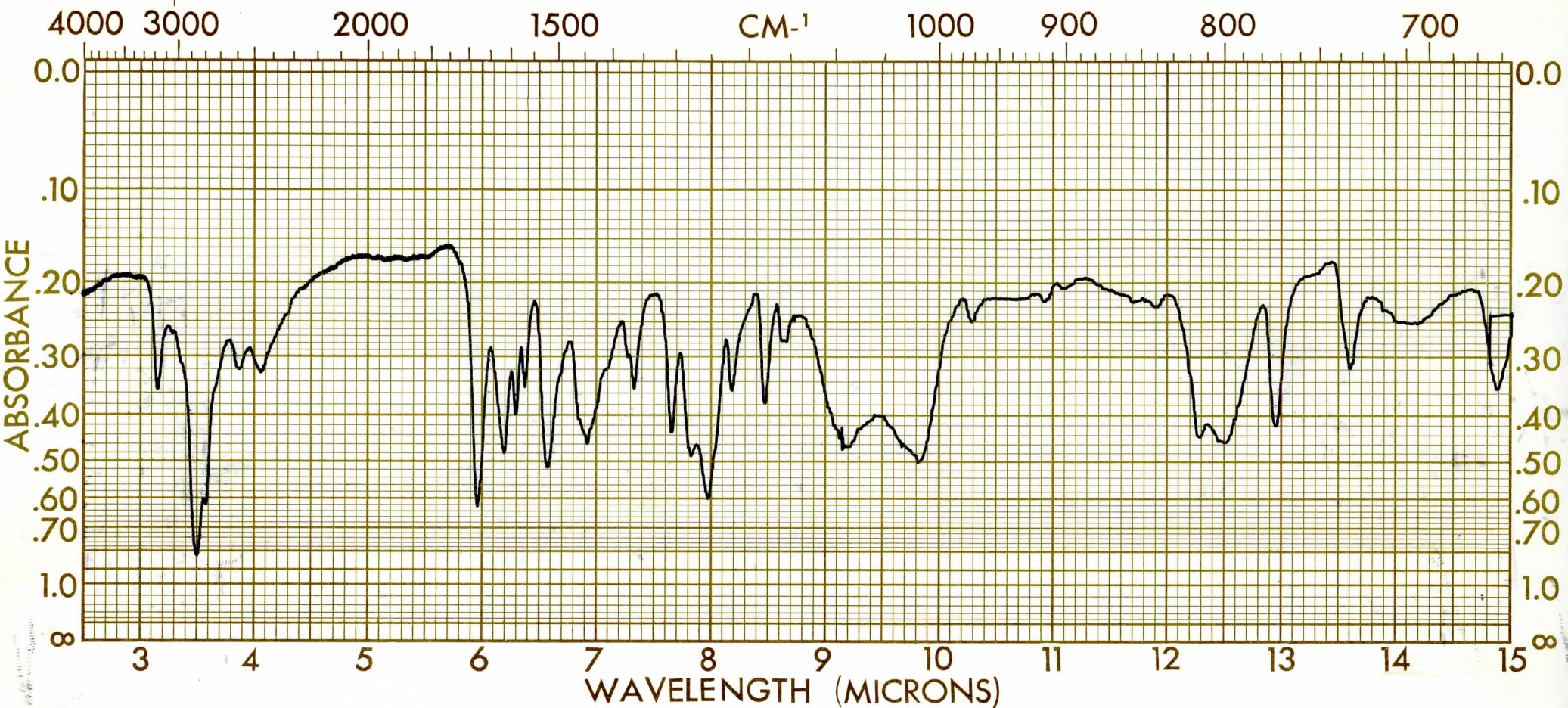
SPECTRUM NO. <u>2</u>	ORIGIN <u>research</u>	LEGEND	REMARKS
SAMPLE	<u>Exp # 556</u>	1.	
<u>2,6 dimethylaniline</u>	PURITY <u>from bottle</u>	2.	
	PHASE <u>liquid cell</u>	DATE <u>12/17/64</u>	
	THICKNESS <u>.0269</u>	OPERATOR <u>J. Scheirer</u>	

SPECTRUM NO. 2
SAMPLE



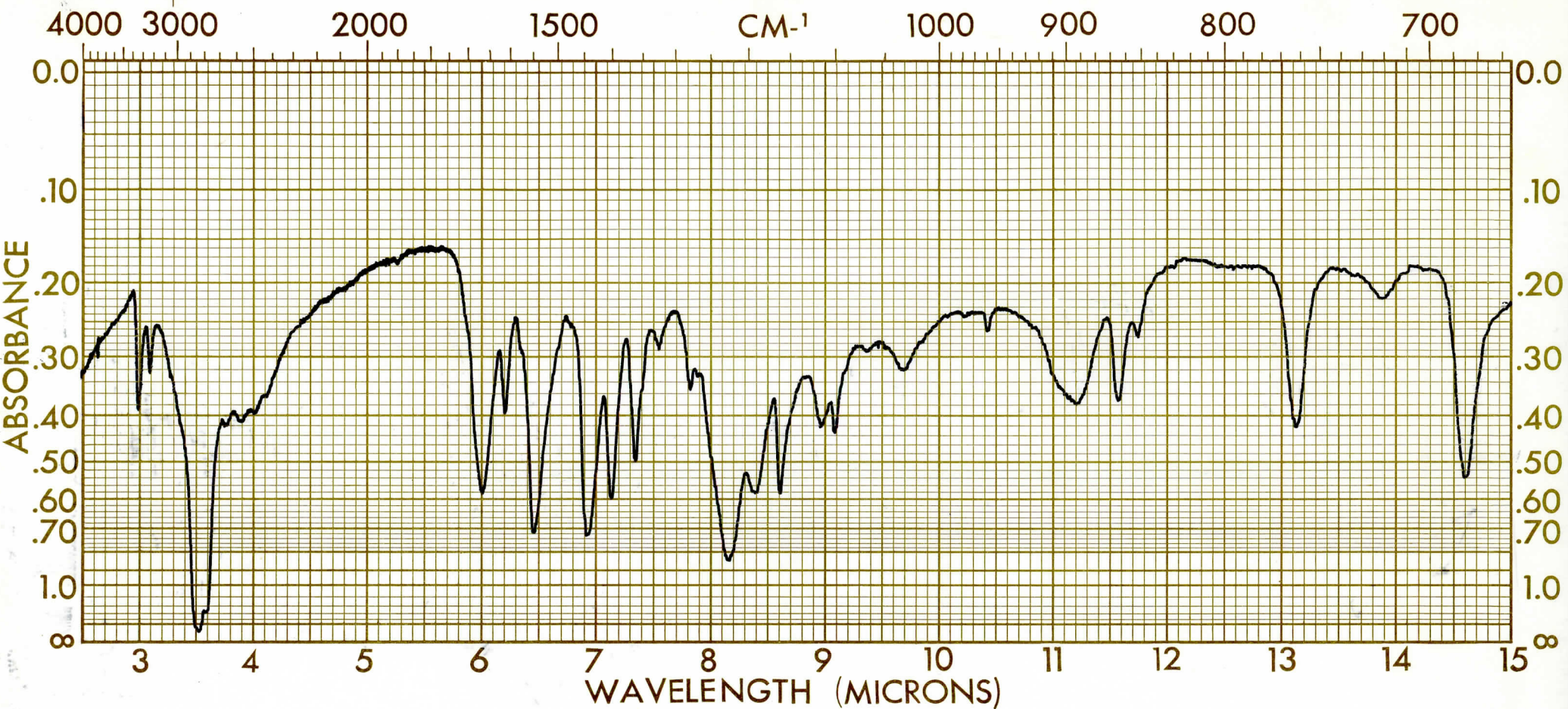
SPECTRUM NO. <u>8</u>	ORIGIN <u>research</u>	LEGEND	REMARKS
SAMPLE <u>2 acetaminophen</u>	<u>Exp A 556</u>	1.	
<u>m-xylene</u>	PURITY <u>R+</u>	2.	
<chem>CC(=O)Nc1ccc(C)cc1</chem>	PHASE <u>solid</u>	DATE <u>2/9/65</u>	
	THICKNESS <u>DMC</u>	OPERATOR <u>J. Scherer</u>	

SPECTRUM NO. _____
SAMPLE



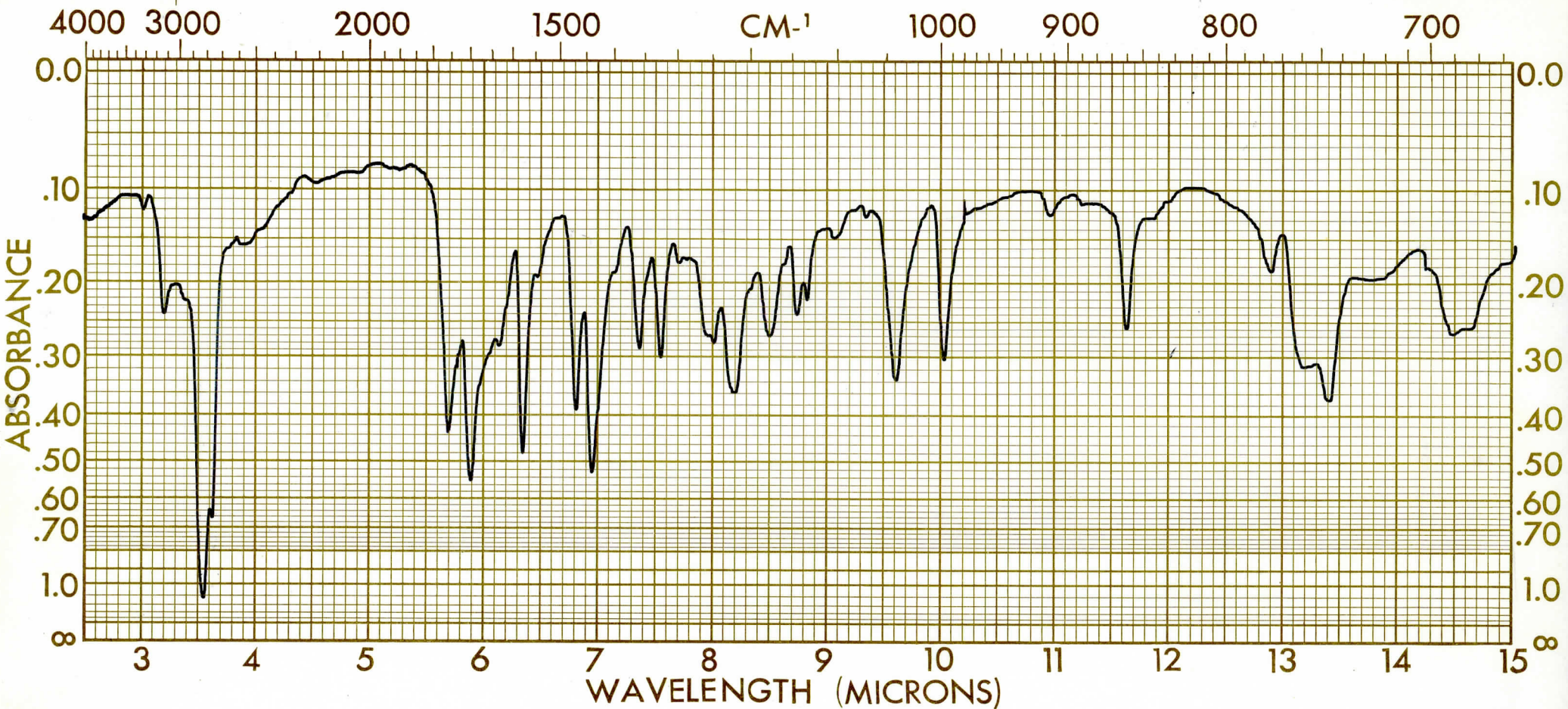
SPECTRUM NO. <u>10</u>	ORIGIN <u>Research</u>	LEGEND	REMARKS
SAMPLE <u>2 acetaminophen</u>	<u>Exp # 556</u>	1. _____	<u>same as 6</u>
<u>isophthalic acid</u>	PURITY <u>2 R X</u>	2. _____	
<u>1200 C</u> <chem>CC(=O)Nc1cccc(C(=O)O)c1</chem>	PHASE <u>mucl</u>	DATE <u>2/9/65</u>	
	THICKNESS <u>0.11 C</u>	OPERATOR <u>J. Scherer</u>	

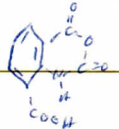
SPECTRUM NO. _____
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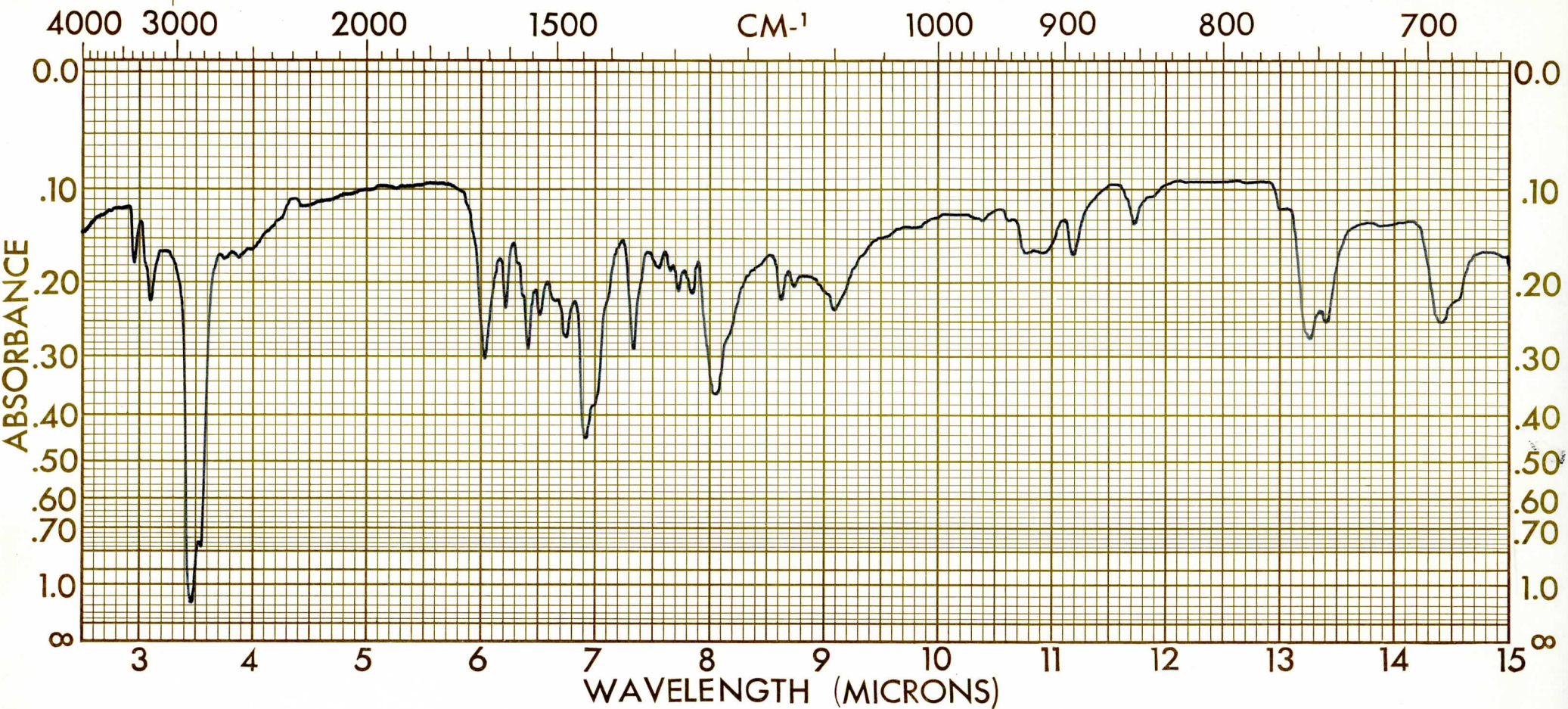
SPECTRUM NO. <u>14</u>	ORIGIN <u>research</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp # 556</u>	1. _____	<u>same as 13</u>
<u>isophthalic acid</u>	PURITY <u>Rx</u>	2. _____	
<chem>NC(=O)c1cccc(c1)C(=O)O</chem>	PHASE <u>Mull</u>	DATE <u>2/23/65</u>	
	THICKNESS <u>DMC</u>	OPERATOR <u>J. Scheiner</u>	

SPECTRUM NO. _____
SAMPLE



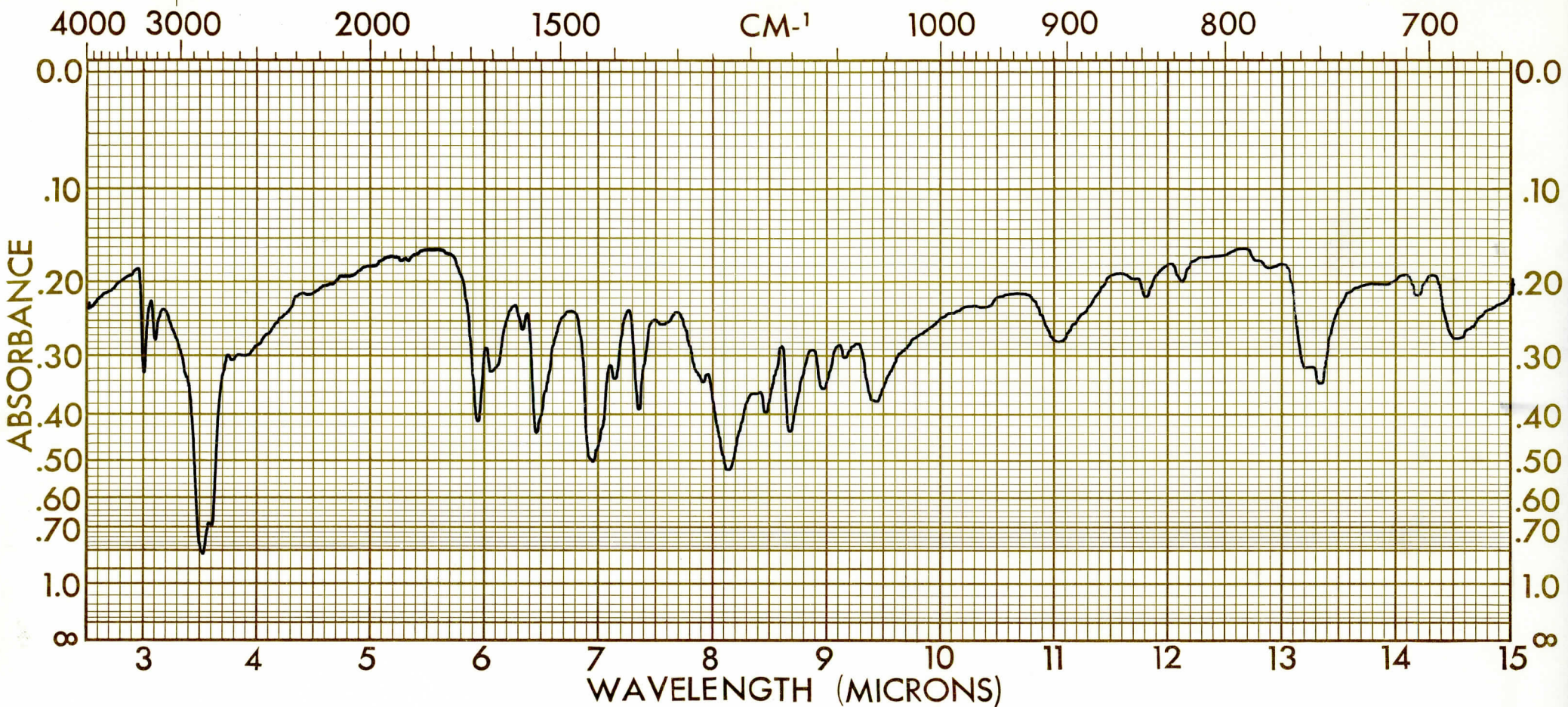
SPECTRUM NO. <u>25</u>	ORIGIN <u>research</u>	LEGEND	REMARKS												
SAMPLE <u>8 carboxy</u>	<u>exp #556</u>	1.	<u>same as # 24</u>												
<u>isatoic anhydride</u>	PURITY <u>2 R +</u>	2.	<u>Analysis Results</u>												
	PHASE <u>Mull</u>	DATE <u>3/16/65</u>	<table><tr><td></td><td><u>theo</u></td><td><u>Found</u></td></tr><tr><td>C</td><td><u>52.18</u></td><td><u>51.96</u></td></tr><tr><td>H</td><td><u>2.43</u></td><td><u>2.58</u></td></tr><tr><td>N</td><td><u>6.76</u></td><td><u>6.54</u></td></tr></table>		<u>theo</u>	<u>Found</u>	C	<u>52.18</u>	<u>51.96</u>	H	<u>2.43</u>	<u>2.58</u>	N	<u>6.76</u>	<u>6.54</u>
	<u>theo</u>	<u>Found</u>													
C	<u>52.18</u>	<u>51.96</u>													
H	<u>2.43</u>	<u>2.58</u>													
N	<u>6.76</u>	<u>6.54</u>													
THICKNESS <u>DNC</u>	OPERATOR <u>J. Scheiner</u>														

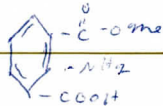
SPECTRUM NO. 25



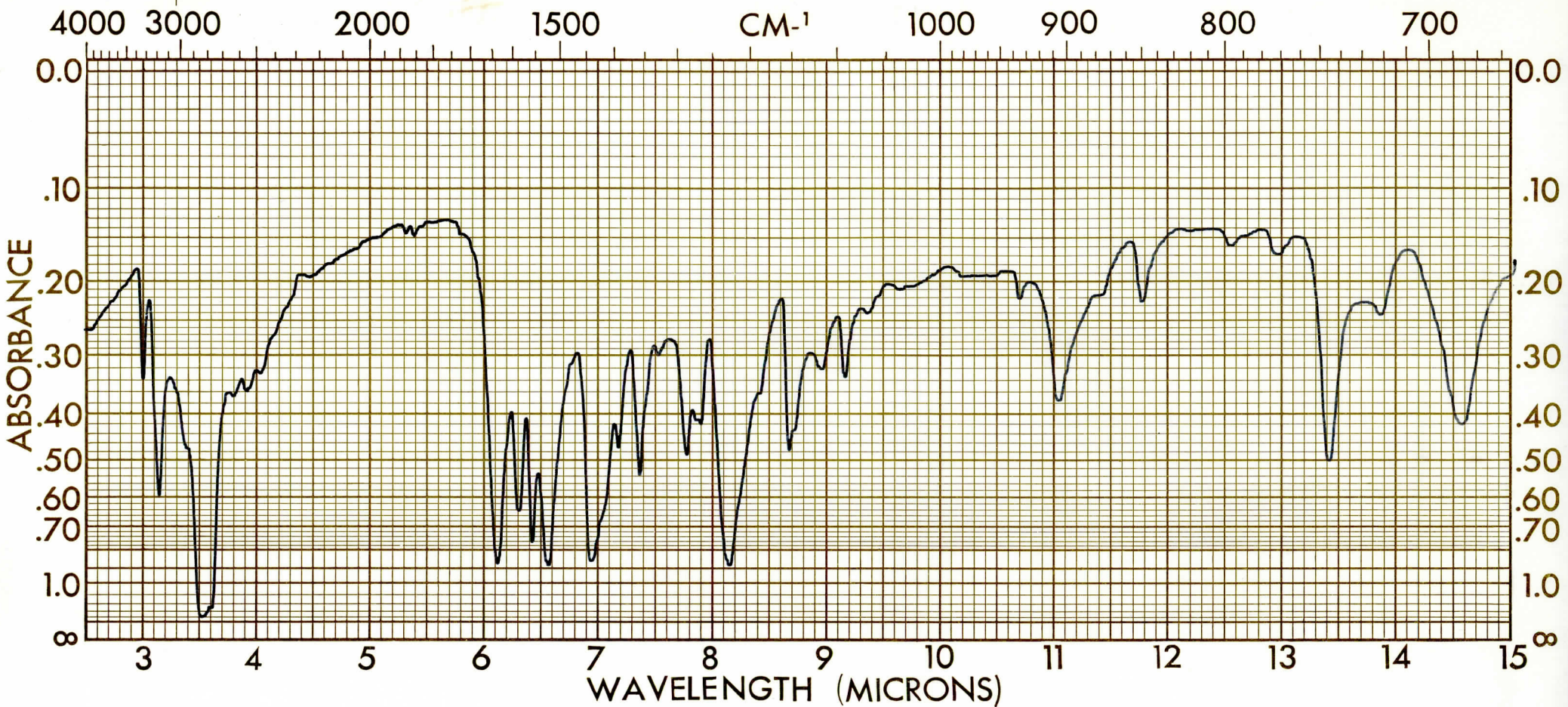
SPECTRUM NO. <u>31</u>	ORIGIN <u>research</u>	LEGEND	REMARKS									
SAMPLE <u>2 amino 3</u>	<u>Exp # 556</u>	1. _____	<u>Analysis Results</u>									
<u>carboxy benzamide</u>	PURITY <u>Q.R.X</u>	2. _____	<table border="1"> <thead> <tr> <th></th> <th>Theo</th> <th>Found</th> </tr> </thead> <tbody> <tr> <td>C -</td> <td>65.63</td> <td>65.43</td> </tr> <tr> <td>H -</td> <td>4.72</td> <td>4.80</td> </tr> </tbody> </table>		Theo	Found	C -	65.63	65.43	H -	4.72	4.80
	Theo	Found										
C -	65.63	65.43										
H -	4.72	4.80										
<div style="text-align: center;"> </div>	PHASE <u>mm 11</u>	DATE <u>3/23/65</u>										
	THICKNESS <u>DMC</u>	OPERATOR <u>J. Scherer</u>										

SPECTRUM NO. _____
SAMPLE _____



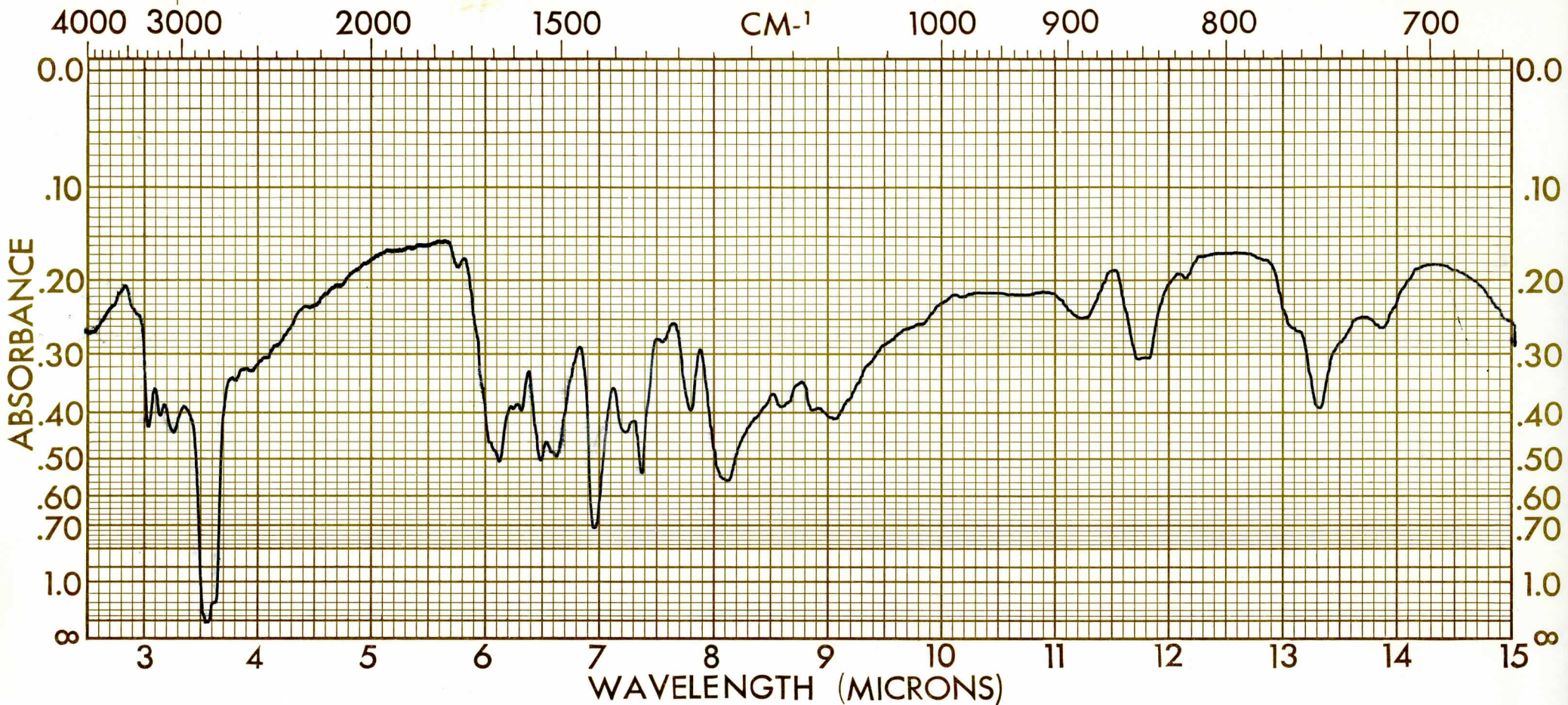
SPECTRUM NO. <u>40</u>	ORIGIN <u>Research</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp # 556</u>	1.	
<u>3 carboxy methyl benzoate</u>	PURITY <u>2 R +</u>	2.	
	PHASE <u>grull</u>	DATE <u>4/22/65</u>	
	THICKNESS <u>DMC</u>	OPERATOR <u>J. Scherer</u>	

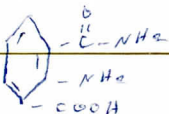
SPECTRUM NO. _____
SAMPLE _____



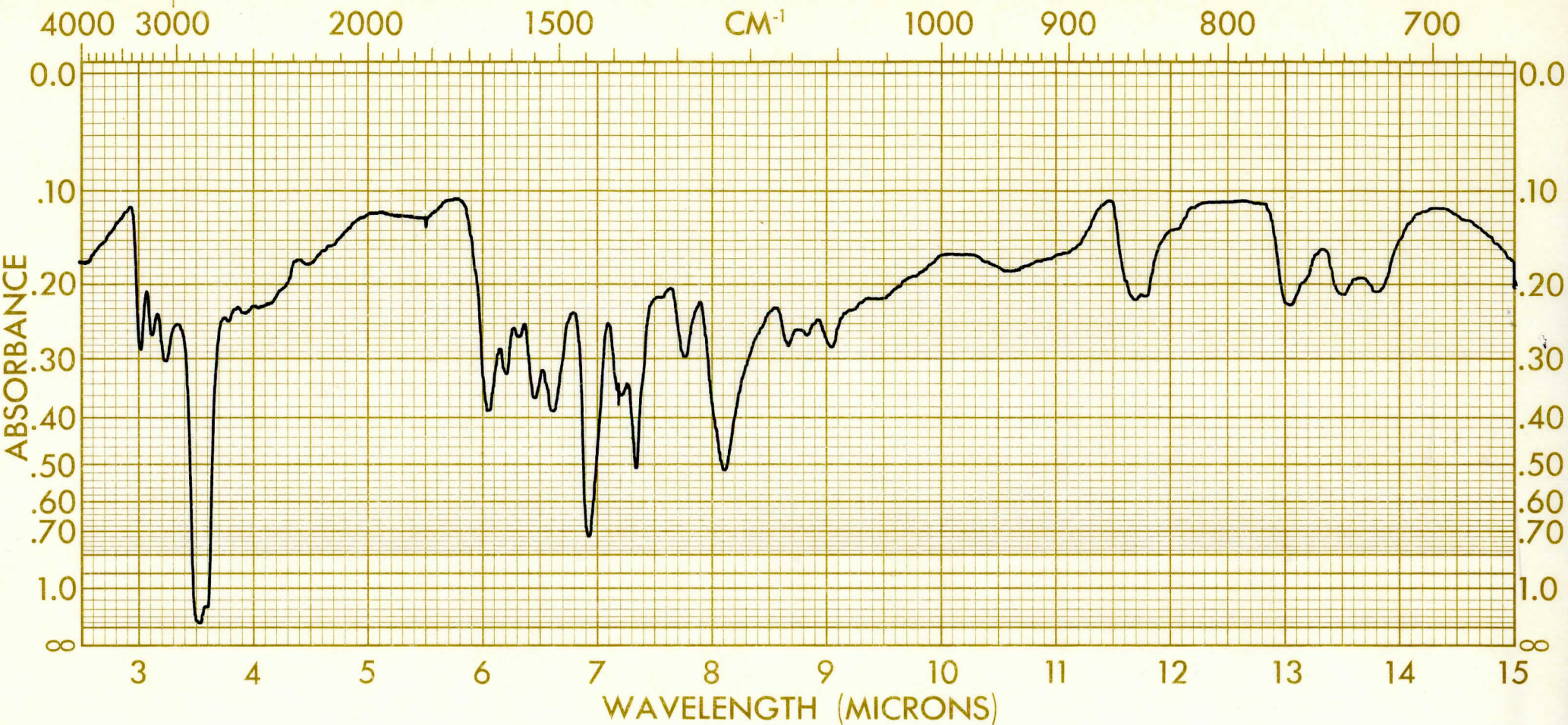
SPECTRUM NO. <u>49</u>	ORIGIN <u>Research</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp # 556</u>	1. _____	
<u>3 carbonyl N hexyl benzamide</u>	PURITY <u>98%</u>	2. _____	
<u>VIII</u> <chem>NC(=O)c1ccccc1C(=O)NCCCCC</chem>	PHASE <u>grind</u>	DATE <u>4/27/65</u>	
	THICKNESS <u>DMC</u>	OPERATOR <u>J. Scherer</u>	

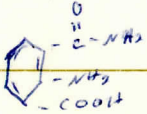
SPECTRUM NO. _____
SAMPLE _____



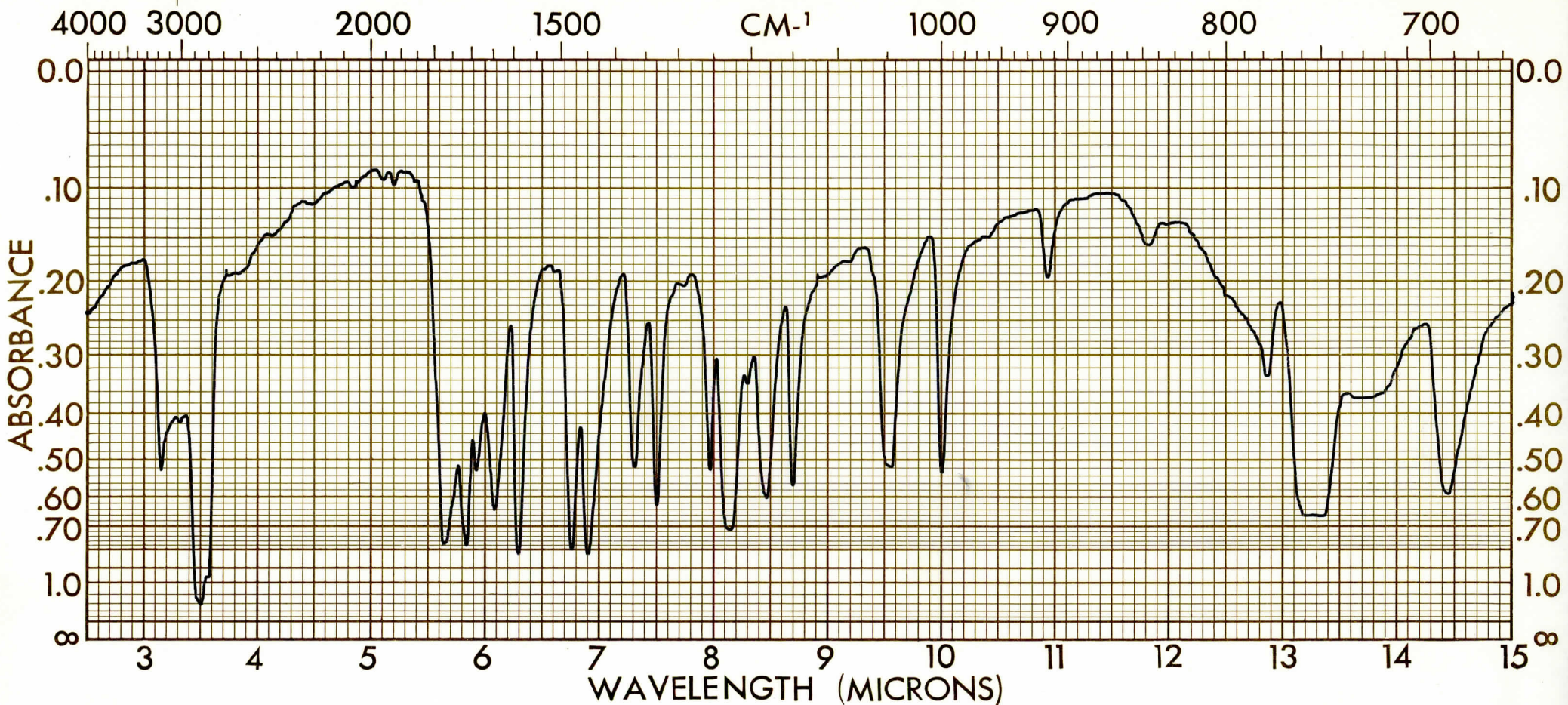
SPECTRUM NO. <u>33</u>	ORIGIN <u>research</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp #556</u>	1. _____	Analysis Results thru Found
<u>isophthalic acid monamide</u>	PURITY <u>2 R +</u>	2. _____	C - 53.33 { 50.16 52.09
	PHASE <u>Mull</u>	DATE <u>4/13/65</u>	H - 4.47 { 4.55 4.65
	THICKNESS <u>DMC</u>	OPERATOR <u>J. J. Rhein</u>	

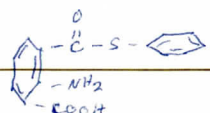
SPECTRUM NO. _____
SAMPLE



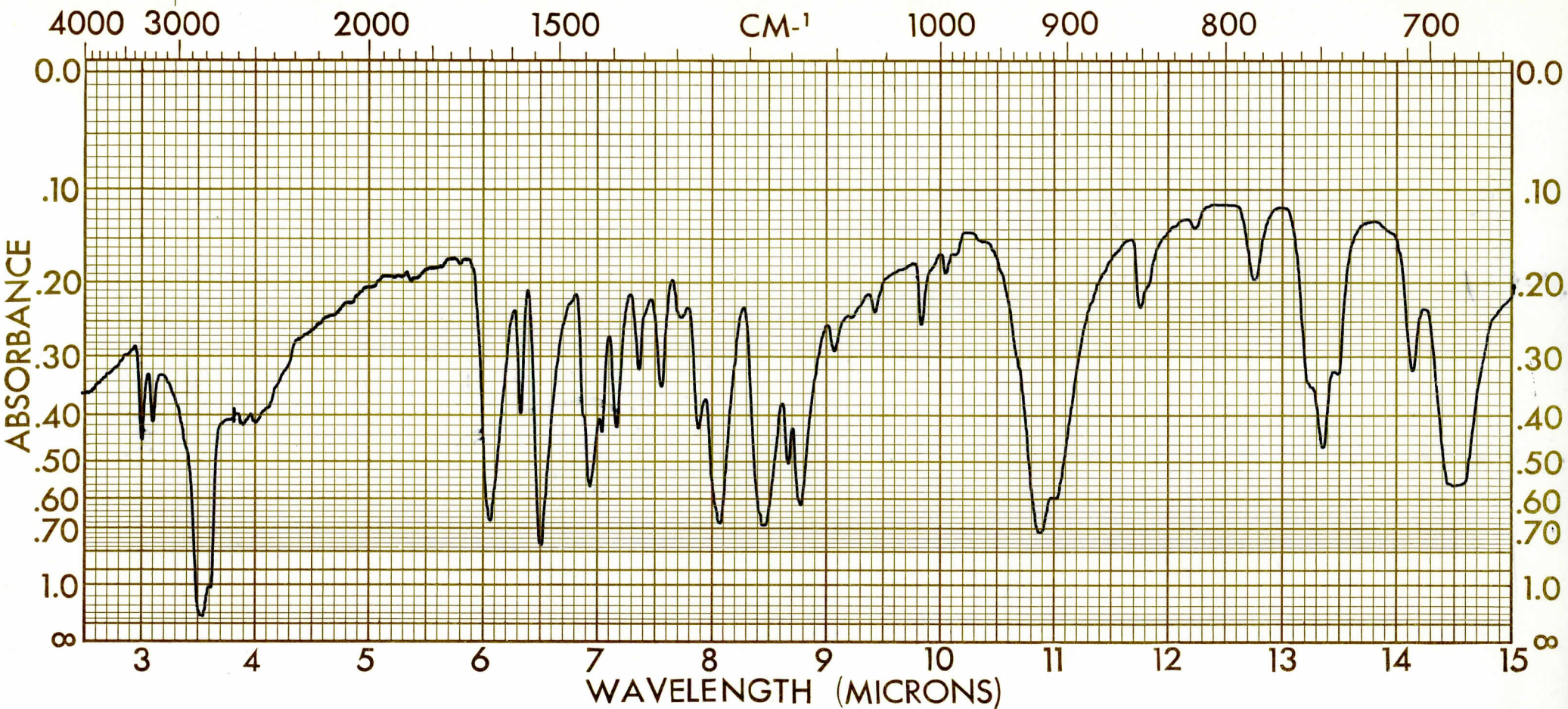
SPECTRUM NO. <u>58</u>	ORIGIN <u>research</u>	LEGEND _____	REMARKS _____
SAMPLE <u>2 amino 3</u>	<u>Exp # 556</u>	1. _____	_____
<u>carboxy benzamide</u>	PURITY <u>2 Rx</u>	2. _____	_____
	PHASE <u>NaCl</u>	DATE <u>4/28/65</u>	_____
	THICKNESS <u>0.01</u>	OPERATOR <u>J. Scherer</u>	_____

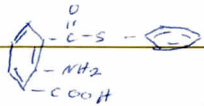
SPECTRUM NO. _____
SAMPLE _____



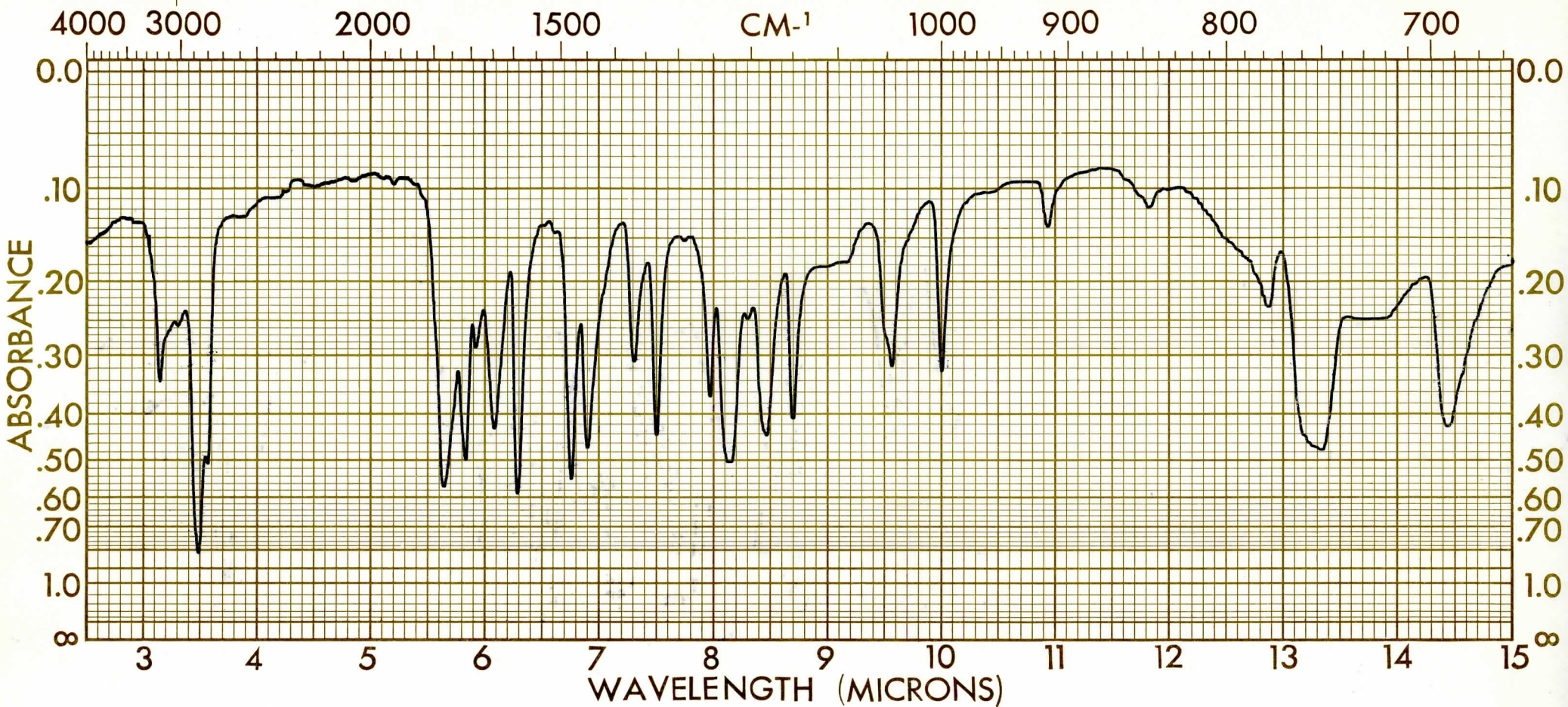
SPECTRUM NO. <u>38</u>	ORIGIN <u>research</u>	LEGEND	REMARKS									
SAMPLE <u>2 amine</u>	<u>Exp # 556</u>	1.	Analysis shows this to be Reclaimed C-3A See I R Graph #25									
<u>3 carbonyl phenyl this benzene</u>	PURITY <u>2 R +</u>	2.	Analysis Results - C-3A									
	PHASE <u>Mull</u>	DATE <u>4/15/65</u>	<table><tr><th></th><th>Theo</th><th>Found</th></tr><tr><td>C</td><td>52.18</td><td>57.83</td></tr><tr><td>H</td><td>2.43</td><td>2.47</td></tr></table>		Theo	Found	C	52.18	57.83	H	2.43	2.47
	Theo	Found										
C	52.18	57.83										
H	2.43	2.47										
THICKNESS <u>DMC</u>	OPERATOR <u>J. Scheiner</u>											

SPECTRUM NO. _____
SAMPLE



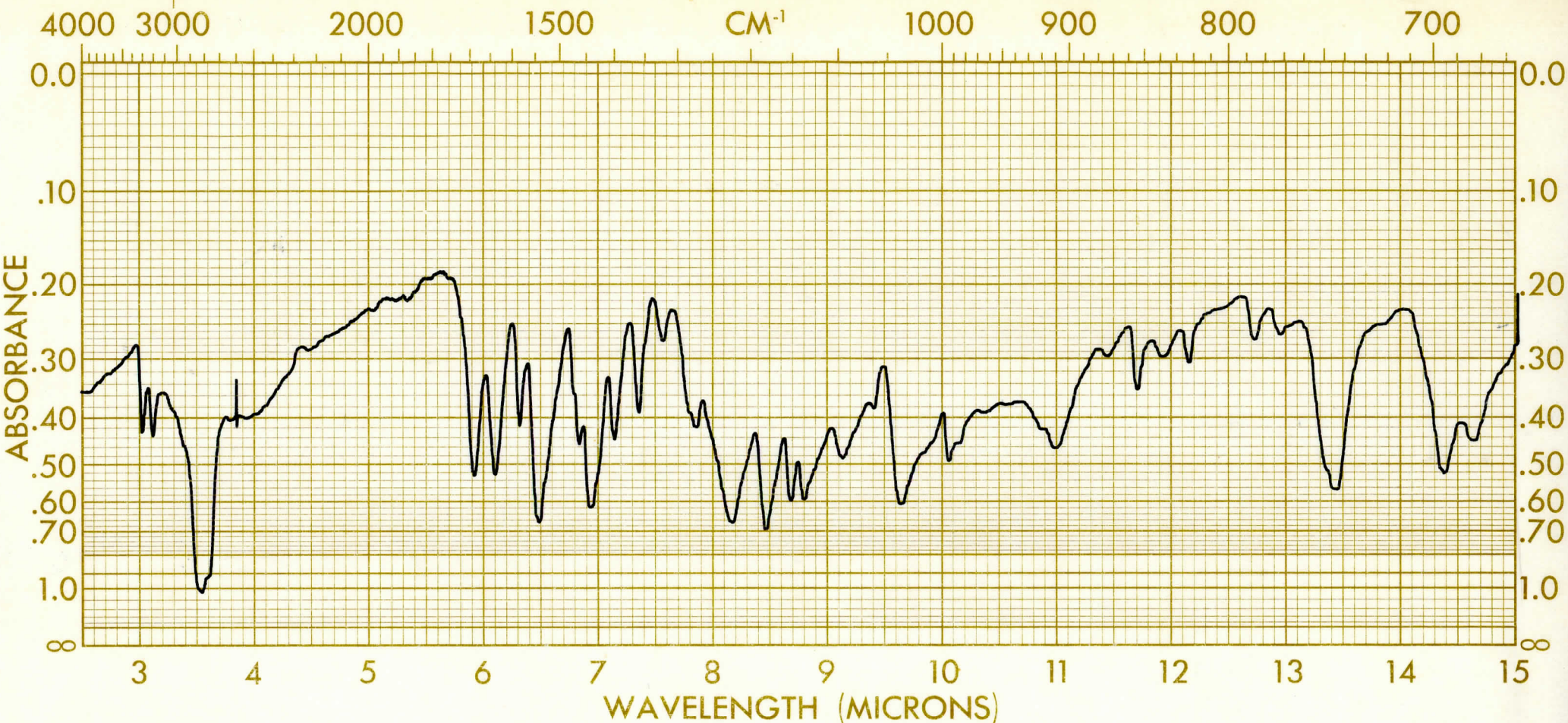
SPECTRUM NO. <u>54</u>	ORIGIN <u>Research</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp # 556</u>	1.	
<u>3-carboxy thiophenyl benzoate</u>	PURITY <u>DR+</u>	2.	
	PHASE <u>Null</u>	DATE <u>4/27/65</u>	
	THICKNESS <u>Dmc</u>	OPERATOR <u>J. S. Baker</u>	

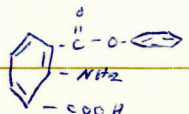
SPECTRUM NO. 54
SAMPLE



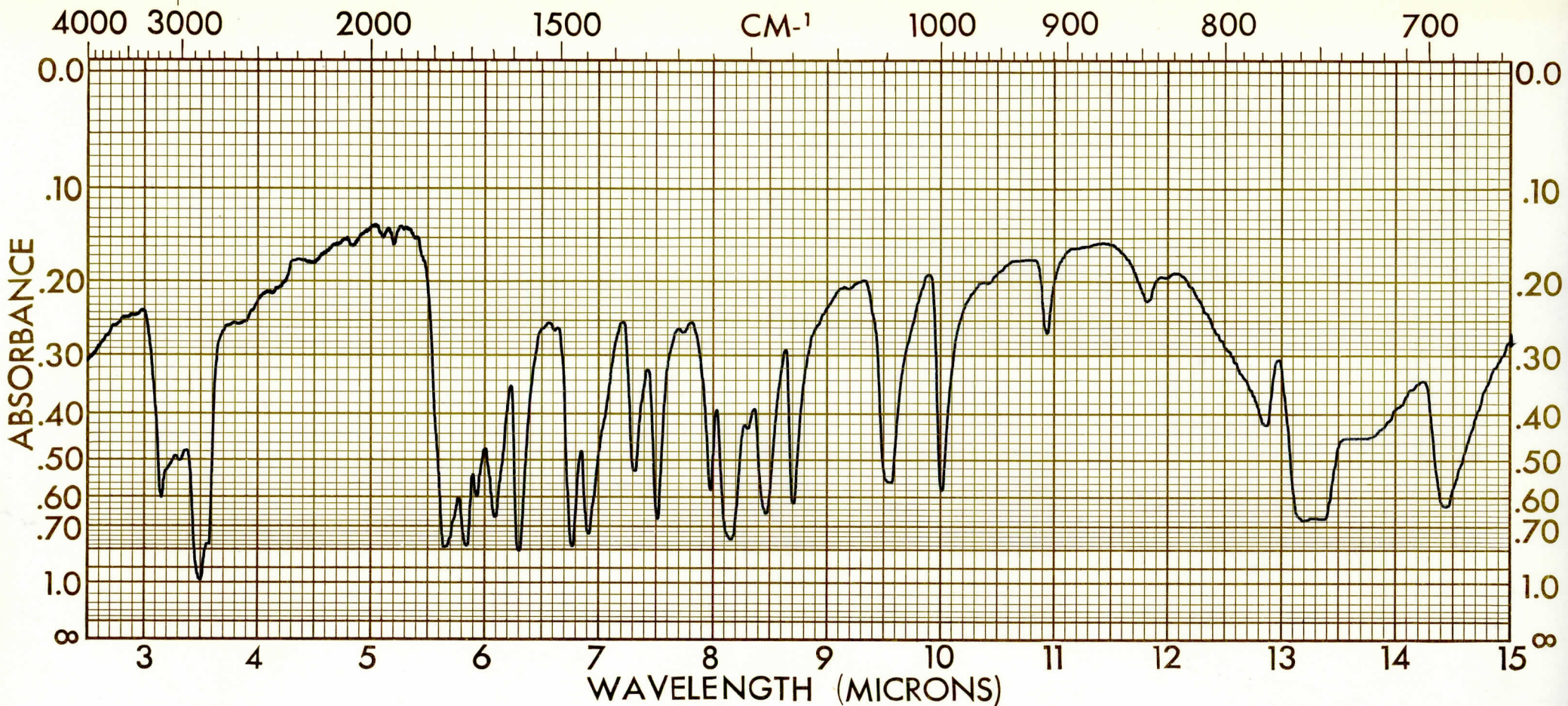
SPECTRUM NO. <u>35</u>	ORIGIN <u>residual</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp # 556</u>	1.	
<u>3 carboxy phenyl benzoate</u>	PURITY <u>100%</u>	2.	
<div style="text-align: center;"> </div>	PHASE <u>solid</u>	DATE <u>4/14/65</u>	
	THICKNESS <u>DMC</u>	OPERATOR <u>J. Scheiner</u>	

SPECTRUM NO. _____
SAMPLE _____



SPECTRUM NO. <u>61</u>	ORIGIN <u>research</u>	LEGEND _____	REMARKS _____
SAMPLE <u>2 amino 3</u>	<u>Exp # 556</u>	1. _____	_____
<u>carboxy phenyl benzoate</u>	PURITY <u>98%</u>	2. _____	_____
	PHASE <u>NaCl</u>	DATE <u>4/29/65</u>	_____
	THICKNESS <u>0.1 cm</u>	OPERATOR <u>J. Scherer</u>	_____

SPECTRUM NO. _____
SAMPLE _____



SPECTRUM NO. <u>45</u>	ORIGIN <u>Research</u>	LEGEND	REMARKS
SAMPLE <u>2 amino</u>	<u>Exp # 556</u>	1. _____	
<u>3 carboxy n-hexyl thio benzamide</u>	PURITY <u>2 R+</u>	2. _____	
<div data-bbox="205 1343 443 1449"> <chem>NC(=O)c1ccccc1SCCCCC(=O)O</chem> </div>	PHASE <u>mucl</u>	DATE <u>4/23/65</u>	
	THICKNESS <u>0.11 c</u>	OPERATOR <u>J. Schires</u>	

SPECTRUM NO. _____
SAMPLE